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(54) Process for producing calcium-supplemented milk drinks

Verfahren zur Herstellung von mit Kalzium angereicherten Milchgetränken

Procédé de préparation de boissons de lait enrichies en calcium

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Description

BACKGROUND OF THE INVENTION

Field of the Invention

[0001] The present invention relates to a process of producing calcium-supplemented milk drinks, and more specifically, the present invention relates to a process of producing calcium-supplemented milk drinks with good flavor and with no precipitation of calcium salts.

Description of the Prior Art

[0002] As the principal component of bone and teeth of animals, calcium is an essential element for organisms. It is required that human adults should take calcium at 1.0 g/day while little children should take it at 0.6 g/day. In recent years in Japan, the shortage of calcium intake has been remarked, which is a serious problem in relation with osteoporosis.

[0003] Nutritionally, cow's milk is essentially a good foodstuff, and if the milk is supplemented with calcium, the calcium shortage can be supplemented by less intake of such milk. The supplementing process in the process of producing calcium-supplemented milk includes the following;

1. a process of adding a soluble calcium salt to cow's milk after heat sterilization, and
2. a process of adding an insoluble calcium salt unreactive with milk protein, such as calcium phosphate and calcium carbonate, followed by heat sterilization of the milk.

[0004] However, the process 1 is disadvantageous in that because the unique unattractive flavor of soluble calcium salts such as calcium chloride affects the taste of cow's milk, only a limited amount of the salts can be added to the milk. Furthermore, because ionized calcium reacts with casein and whey protein to consequently decrease the heat resistance of the milk, a sterilization process to prepare a commercially aseptic state is never applicable to the milk. Thus, the milk cannot be prepared into a long-life type. It is known a process comprising individually sterilizing milk base and calcium under heating and subsequently mixing them together, but the procedures of the process are laborious. Because calcium ions promote the modification of protein into unstable protein and promote also the precipitation thereof even at lower temperatures, furthermore, the amount of calcium ions to be added to the milk is limited so that the milk cannot be supplemented with a satisfactory amount of calcium.

[0005] Alternatively, the process 2 is problematical in that rough feeling may be caused in the milk unless a particle size of the insoluble calcium salts is not considerably small and in that the calcium salts may readily precipitate because of the insolubility. So as to overcome the problems of the insolubility, various processes have been suggested, for example, a process comprising adding 0.01 to 0.06 % by weight of a stabilizer such as carrageenan and gum guar to cow's milk (Japanese Patent Laid-open No. Sho 62-248450): a process comprising depositing insoluble calcium salts in fine particles onto fat spheres in the emulsion of a fat and water or comprising embedding the salts into the fat spheres in the emulsion to adjust the specific gravity of these particles approximately to the specific gravity of the liquid phase (Japanese Patent Laid-open No. Sho 57-110167): a process comprising adding crystalline cellulose to the milk to prepare a fine network structure in the liquid phase, thereby supporting the insoluble calcium salts in fine particles to prevent the precipitation thereof (Japanese Patent Publication No. Sho 57-35945): or a process comprising mixing calcium carbonate in slurry with an aqueous solution of a hydrophilic emulsifier, and subsequently subjecting the mixture solution to a dehydration process, prior to vacuum drying, whereby the aggregation can be prevented (Japanese Patent Laid-open No. Sho 63-173556). However, all of the processes have disadvantages of the modification of the properties of cow's milk of itself, the calorie increase of cow's milk which is not suitable for low-fat milk, or the escalation of the production cost.

[0006] Because it is difficult to prevent the precipitation of general calcium salts, a process of supplementing cow's milk with calcium is suggested, comprising supplementing milk with fine particle calcium phosphate or calcium citrate which is recovered from whey as a byproduct of cheese production, characterized in that no precipitation occurs (Japanese Patent Laid-open No. Sho 60-248152). These calcium salts are in a network structure so the salts can keep their stable state with nearly no generation of the precipitate in solution. Additionally, the calcium salts are not water-soluble because of their non-ionic state, so that the salts do not react with protein and no precipitation occurs. Because the salts are not crystalline, their network structure can be maintained as it is. The salts never crystallize to cause precipitation.

[0007] Since the network structure of itself of the calcium salts secondarily binds together to be modified into a polymerized state, the salts may therefore precipitate consequently. So as to prevent such precipitation, it is required

to preliminarily mix the salts with fat.
Due to the consequent calorie increase, the resulting milk is not suitable as low-fat milk, disadvantageously ; or the production cost is elevated, disadvantageously.

[0008] EP-A-0408756 discloses a process for preparing a milk mineral concentrate from whey which can be added to various drinks, for instance cow's milk. That process comprises (i) ultrafiltering whey at pH 4-6, (ii) concentrating the filtrate thus obtained until the lactose concentration reaches about 50 %, (iii) allowing the concentrate to stand (at 0-20°C for 10-12 h) then (iv) removing the precipitated lactose. Example 11 of EP-A-0408756 describes the obtention of a mixture from cow's milk and said milk mineral concentrate. Since its ultrafiltration is performed at pH 4-6 for recovering the filtrate, EP-A-0408756 does neither teach nor suggest the process if this invention which comprises in particular a filtration technique at pH 6-9 for recovering the solid product (retentate or precipitate).

[0009] The paper by B. WEBB et al., "Byproducts from milk", THE AVI PUBLISHING COMPANY INC., Westport, Connecticut, 1970, pages 363-369, is concerned with the isolation of lactose from milk and discloses (see page 369) a process comprising (i) recovering the mother liquor obtained during the isolation of lactose from whey, (ii) optionally drying said mother liquor, (iii) adding the product (i) or (ii) thus obtained to food for instance skimmed milk, (iv) drying the resulting mixture, then using the dried product thus obtained as food or as food additive for human beings or animals.

[0010] DE-A-4113836 discloses a process comprising (i) providing a sweet whey permeate obtained at pH 6.8-7.2 and having a dry matter content of 5-15 % by weight (see page 2 line 19), (ii) evaporating said permeate at pH 6.8-7.2 to obtain a concentrated permeate having a dry matter content of 30-60 % by weight, (iii) heating said concentrated permeate at a temperature of at least 80°C for 15-45 minutes [preferably at least 90°C for 30-45 minutes (see page 2 lines 61 and 63-64)] in order to precipitate a (lactose/protein/mineral salt) solid mixture I, (iv) discarding said mixture I, (v) contacting the recovered filtrate with active carbon at 45-95°C then discarding the active carbon by filtration, (vi) concentrating the discoloured filtrate thus obtained to precipitate crystalline lactose, (vii) separating the crystalline lactose from the remaining solution, and (viii) microfiltering said remaining solution to separate a (lactose/protein/mineral salt) solid mixture II from the filtrate. Said solid mixture is useful as an additive in the nutrition, beverage, filling and fermentation fields. However that process for obtaining said (lactose/protein/mineral salt) solid mixture I, which has a calcium content of 7.15 % (according to Ex. 1), does compulsory imply performing a drastic heat treatment of at least 80°C (90°C for 30-45 minutes, according to example 1) on the pH-adjusted concentrated permeate after ultrafiltration of whey to form a suspension. Such a heat treatment at a high temperature is not carried out in the present invention.

[0011] Those three publications do neither disclose nor suggest the important features of the present invention which are :

(α) preparing a whey mineral from whey so that the whey mineral can have a calcium content of 2-8 % by weight,

(i) as a retentate in ultrafiltration or microfiltration or
(ii) as a precipitate in centrifugation,

at pH 6.0-9.0, and
(β) adding the whey mineral thus obtained to cow's milk in order to form a calcium-supplemented cow's milk drink so that the calcium supplied by said whey mineral can be 10-40 mg % therein.

Problems to be Solved by the Invention

[0012] Thus, it is an object of the present invention to provide a process of producing a calcium-supplemented milk dring without the aforementioned drawbacks.

Means for Solving the Problems

[0013] The prevent inventors have found that by adding to cow's milk or the like, a whey mineral being generated from whey and containing a good balance of insoluble calcium salts and soluble calcium salts and additionally containing a good balance of mineral other than calcium, the flavor of the milk of itself is improved with no precipitate of the calcium salts, whereby a long-life type of milk can be prepared. Thus, the present invention has been achieved.

[0014] More specifically, the present invention is to provide a process of producing a calcium-supplemented milk drink, comprising adding the whey mineral at a calcium content of 2 to 8 % by weight to cow's milk or the like. Subject of the invention

[0015] This invention is concerned with a process for producing a calcium-supplemented cow's milk drink, said process, comprising using whey as a Ca source, obtaining a mixture of whey and cow's milk then sterilizing said mixture.

[0016] According to a first aspect of this invention, said process is characterized in that it comprises the following

steps :

(a) whey is subjected to protein removal and lactose removal,
(b) the resulting whey is subjected to pH adjustment to 6.0-9.0,
(c) the resulting whey is further subjected to ultrafiltration or microfiltration to obtain a concentrate,
(d) the concentrate thus obtained is subjected to drying to obtain a whey mineral having a calcium content of 2-8 % by weight,
(e) the whey mineral thus obtained is added to cow's milk to form a calcium-supplemented cow's milk drink so that the amount of calcium supplied by the whey mineral represents 10-40 mg % therein (10-40 mg Ca for 100 g calcium-supplemented cow's milk drink), and
(f) the calcium-supplemented cow's milk drink thus obtained is subjected to pH adjustment to 6.0-7.5.

[0017] According to a second aspect of this invention, said process is characterized in that it comprises the following steps :

(a) whey is provided,
(b) the whey thus provided is subjected to protein removal,
(c) the resulting whey is subjected to pH adjustment to 6.0-9.0,
(d) the resulting whey is further subjected to centrifuging to obtain a precipitate,
(e) the precipitate thus obtained is subjected to drying to obtain a whey mineral having a calcium content of 2-8 % by weight,
(f) the whey mineral thus obtained is added to cow's milk to form a calcium-supplemented cow's milk drink so that the amount of calcium supplied by the whey mineral represents 10-40 mg % therein (10-40 mg Ca for 100 g calcium-supplemented cow's milk drink), and
(g) the calcium-supplemented cow's milk drink thus obtained is subjected to pH adjustment to 6.0-7.5.

[0018] According to a third aspect of this invention, said process is characterized in that it comprises the following steps :

(a) whey is provided,
(b) the whey thus provided is subjected to pH adjustment to 6.0-9.0,
(c) the resulting whey is further subjected to centrifuging to obtain a precipitate,
(d) the precipitate thus obtained is subjected to drying to obtain a whey mineral having a calcium content of 2-8 % by weight,
(e) the whey mineral thus obtained is added to cow's milk to form a calcium-supplemented cow's milk drink so that the amount of calcium supplied by the whey mineral represents 10-40 mg % therein (10-40 mg Ca for 100 g calcium-supplemented cow's milk drink), and
(f) the calcium-supplemented cow's milk drink thus obtained is subjected to pH adjustment to 6.0-7.5.

[0019] In each of these three aspects, the calcium-supplemented cow's milk drink thus obtained is heat resistant with no precipitation of Ca salts and stands sterilization as a final step.

[0020] The present invention will be described in detail.

[0021] Whey mineral can be produced from whey such as cheese whey, for example Gouda cheese whey, cheddar cheese whey and the like ; and acid whey for example lactate whey, hydrochloride whey and the like.

[0022] Protein can be removed from whey by routine processes (deproteination), for example by the use of ion exchangers and by means of filtration and concentration. More specifically, such a process comprises mixing whey with a cation exchanger thoroughly whashed with an alkali such as 0.1N sodium hydroxide and subsequently with desalted water, and agitating the resulting mixture. Then, the resulting mixture is preferably adjusted to pH 2 to 5 by using an acid such as 6N sulfuric acid. Subsequently, the cation exchanger is filtered off, and the resulting filtrate is concentrated to a final solid content of about 40 to 60 %, by using a rotary evaporator or the like.

[0023] The deproteination of whey may also be conducted by ultrafiltration. The ultrafiltration for such deproteination comprises solubilizing the calcium salts in whey, preferably under conditions of acidity (pH 6 or less), so as to recover the product in the pass-through fraction. The deproteination may be carried out by microfiltration, but ultrafiltration is more advantageous in terms of the efficiency of deproteination.

[0024] Lactose may be removed from whey by routine processes, including for example a process comprising adding lactose to whey to crystallize the lactose therein and then filter off the crystallized lactose. Preferably, lactose in a fine preparation should be added to whey. Lactose may be crystallized by routine processes, including for example a process comprising adding seed -lactose at a ratio of 0.01 to 0.1 % to the solids of a whey solution adjusted to a solid

concentration of 40 to 60 %, subsequently stirring the mixture solution gently and allowing the solution to stand at 0 to 7 °C for 10 hours or more.

[0025] Calcium may be concentrated at a pH of 6.0 to 9.0 by ultrafiltration, microfiltration or the like. Whey should be concentrated by ultrafiltration or microfiltration, so that the final calcium content in the whey mineral might be of 2 to 8 % by weight, preferably 4 to 6 % by weight. If the content is less than 2 % by weight, the ratio of the blended whey mineral to cow's milk or the like is so elevated that the heat resistance of the resulting product possibly may be deteriorated under the influence of the minerals other than calcium. If the content is more than 8 % by weight, calcium may possibly precipitate during the storage of the resulting product for a short term. If whey mineral at a desired calcium level cannot be generated by a single process of ultrafiltration or microfiltration, the process may satisfactorily be repeated.

[0026] The whey mineral at a calcium content of 2 to 8 % by weight may be generated by centrifuging. For example, by adjusting whey deproteinized via a cation exchanger or untreated whey to pH 6 to 9 and then centrifuging the pH-adjusted whey at 10,000 g for about 10 minutes, calcium can be recovered together with aggregated protein and complexes of protein with lipids. Alternatively, by adjusting the calcium concentration and pH of whey to desired levels (pH adjusted to 6,0-9,0) and subsequently heating gently the whey prior to centrifuging, a calcium fraction may be recovered similarly (Bulletin of the International Dairy Federation 212, Chapter 24, Session VI, p. 154, 1987).

[0027] The whey mineral thus recovered contains well-balanced insoluble calcium salts and soluble calcium salts, and contains additionally minerals other than calcium, such as potassium, sodium, magnesium and the like.

[0028] In accordance with the present invention, the whey mineral described above is added to cow's milk. Cow's milk include cow's milk, specific cow's milk, partially defatted milk, defatted milk (skimmed milk), defatted powdery milk, reconstituted milk, processed milk, milk drink and the like ; and for defatted powdery milk, for example, the whey mineral and defatted powdery milk may be added to water. The whey mineral is then added in such an amount that the content of the calcium derived from the whey mineral is 10 to 40 mg %, preferably 20 to 30 mg % in the resultant calcium-supplemented milk drink (the unit "mg %" represents a content in mg of the supplemented calcium containent in 100 g of the resultant calcium-supplemented milk drink). When the amount thereof to be added is more than 40 mg %, the heat resistance may be deteriorated or calcium may possibly precipitate.

[0029] The whey mineral added to cow's milk or the like should preferably be homogenized by routine processes. The homogenizing processes may be carried out by means of a homogenizer for general use. The homogenization may preferably be conducted at 40 °C or less. When the homogenization is carried out at higher temperatures, free calcium may potentially make casein unstable so the aggregation of casein is eventually observed before the sterilization process described below. When the homogenization is carried out at a low temperature of 40 °C or less, stable complexes of casein, and the whey protein and the calcium of the whey mineral may be formed to possibly put the casein at a relatively stable state before the sterilization process or during the process.

[0030] The calcium-supplemented milk drink thus produced is adjusted to pH 6.0 to 7.5, specifically pH 6.5 to 7.2. Through the pH adjustment, the deterioration of the heat resistance, the browning during heating or the decomposition of the protein can be prevented. Under general conditions, the pH of the resulting product should be within the range described above, but if necessary, the pH adjustment may be done using sodium hydroxide, potassium hydroxide or the like.

[0031] By sterilizing the thus produced calcium-supplemented milk drink to a substantially sterile state (a commercially aseptic state) and aseptically filling the milk in containers or the like, the milk drink may be prepared into a calcium-supplemented long-life milk drink, which can be stored even at ambient temperature for several months. The sterilization may be carried out satisfactorily by routine processes, for example a process of continuous heating at ultra-high temperatures (UHT) under the conditions of 130 to 150 °C for 1 to 10 seconds. If the calcium content of the whey mineral is preliminarily adjusted to 2 to 8 % by weight by the process of the present invention, the heat resistance can be maintained well. Hence, a relatively long time will be needed even if the sterilization is carried out as described above, until the aggregation initiates. Thus, no scorching of the protein onto sterilizing machines, no precipitation of the sterilized solutions, or no formation of aggregates may occur.

[0032] Because calcium is supplemented by the use of the whey mineral in the calcium-supplemented milk drink produced by the process described above in accordance with the present invention, the milk drink has good flavor with no precipitation of calcium salts or without rough feeling. Such good flavor is understood from the fact that whey mineral is used as an alternative seasoning to table salt; and is due to the fact that the whey mineral is a co-precipitate of the mineral components with the residual phospholipids and other fat-soluble components in the whey. Thus, by adding the aforementioned whey mineral into cow's milk or the like, specifically a low-fat milk prepared by using a partially defatted milk, a defatted milk (skimmed milk), a defatted powdery milk or the like as the principal raw material, the resulting milk can procure body similar to the original body of milk fat, together with good flavor similar to the flavor of cow's milk. Because the milk drink pass through high-temperature sterilization processes, the milk drink is prepared into a calcium-supplemented long-life milk drink.

[0033] The present invention will now be described in examples, but the scope of the present invention is not limited

to these examples.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Production Example 1

[0034] Pouring 20 liters of Gouda cheese whey into 4 liters of a cation exchanger (Indion S3, manufactured by Life Technology Inc.) washed with 0.1N NaOH and thoroughly washed with desalted water, the mixture was agitated in a tank to be adjusted to pH 3.0 with 6N H_2SO_4. Subsequently, the cation exchanger was filtered off. The resulting filtrate was concentrated to a final solid content of 40 % by means of an evaporator (the thus-obtained product is referred to as "a partially deproteinized solution").

[0035] Adding 1 g of finely ground lactose to the concentrated solution, the mixture was then agitated at 2 °C for 18 hours to crystallize the lactose therein. The crystallized lactose was filtered off, and the resulting filtrate was freeze-dried, to recover a composition shown in Table 1 (Whey mineral No.1) at a yield of 350 g.

Table 1

Whey mineral No.1	
Water content	2.0 % by weight
Protein	14.5 % by weight
Ash content	30.2 % by weight
Fat	4.2 % by weight
Carbohydrates*	49.1 % by weight
Calcium	1.4 % by weight.

* balance to 100 %

Production Example 2

[0036] Adjusting a partially deproteinized solution recovered in the same manner as in the Production Example 1 to pH 8.0 with 6N NaOH, the resulting solution was concentrated by 10 fold with an ultrafiltration membrane of a fractional molecular weight of 20,000 (LAB-20.0.72, GR61PP of a membrane area of 0.36 m^2, manufactured by DDS, Co. ,Ltd.). After adding furthermore desalted water of a 10-fold volume that of the concentrated solution, the resulting solution was concentrated again by 10 fold at pH 8.0. By freeze-drying then the resulting concentrated solution, a composition shown in Table 2 (Whey mineral No.2) was recovered at a yield of 100 g.

Table 2

Whey mineral No.2	
Water content	2.0 % by weight
Protein	51.9 % by weight
Ash content	29.9 % by weight
Fat	12.3 % by weight
Carbohydrates*	3.9 % by weight
Calcium	4.2 % by weight

* balance to 100 %

Production Example 3

[0037] Concentrating Gouda cheese whey (1,000 kg) by 20 fold at pH 6.0 by using the same ultrafiltration membrane as in the Production Example 2, and concentrating the pass-through solution with an evaporator, lactose was crystallized and removed off in the same manner as in the Production Example 1. Diluting the mother solution with desalted water to a final solid content of 10% and adjusting the resulting solution to pH 8.0 with 6N NaOH, the solution was again subjected to an ultrafiltration procedure. At concentration ratios of 5, 6, 7 and 10, some of the individual concentrated solutions recovered were freeze-dried, to generate compositions shown in Table 3 (Whey minerals Nos. 3-1, 3-2, 3-3, and 3-4, at concentration ratios of 5, 6, 7, and 10, respectively).

Table 3

Components	Whey mineral 3-1 (UFx5)	Whey mineral 3-2 (UFx6)	Whey mineral 3-3 (UFx7)	Whey mineral 3-4 (UFx10)
Water content	1.5	2.0	2.0	1.8
Protein	10.1	16.6	18.0	22.1
Ash content	24.0	22.2	21.8	20.5
Fat	0.3	0.8	0.9	1.2
Carbohydrates*	64.1	58.4	57.3	54.4
Calcium	2.4	6.6	7.4	9.6

* balance to 100 % (unit; % by weight)

Production Example 4

[0038] Adjusting Gouda cheese whey (500 kg) to pH 7.2, and allowing the whey to stand at 55 °C for 10 minutes, the resulting whey was supplied at a feed of 50 kg/h into a centrifuge (LAPX202, manufactured by α-Laval Co., Ltd.) at 80,000 rpm. Drawing out the precipitate every one hour and freeze-drying the precipitate, a composition shown in Table 4 (Whey mineral No.4) was recovered at a yield of 1.8 kg.

Table 4

Whey mineral No.4	
Water content	4.1 % by weight
Protein	15.7 % by weight
Ash content	16.6 % by weight
Fat	3.8 % by weight
Carbohydrates*	59.8 % by weight
Calcium	2.7 % by weight

* balance to 100 %

Example 1

Testing of heat resistance

[0039] Dissolving a defatted powdery milk (10.3 g) (manufactured by Meiji Milk Products, Co., Ltd.) in a small volume of desalted water, adding one of the whey minerals produced in the Production Examples 1 to 4 (Whey Mineral Nos. 1, 2, 3-1, 3-2, 3-3, 3-4, and 4) to the solution to a final calcium content of 20 to 50 mg, desalted water was further added to the solution to a final total weight of 100 g. The mixture solution was then agitated and mixed together with an Excel auto-homogenizer (manufactured by Nippon Seiki, Co., Ltd.) at 10,000 rpm for 3 minutes, to prepare a sample for the testing of heat resistance.

[0040] Using a shaking-type heat resistance tester (manufactured by Ishiyama Scientific Machinery Industries), the test of heat resistance was carried out at an oil-bath temperature of 135 °C (or 140 °C). The heat resistance was shown as the time (CT in unit minute) required for the coagulation firstly observed in a sample solution. All the sample pHs were within a range of 6.6 ± 0.1. The results are shown in Table 5.

Table 5

Relation between calcium level derived from whey mineral and coagulation time (CT in unit minute)								
Ca level from whey mineral	Control $CaCl_2$	Whey mineral No.1	Whey mineral No.2	Whey mineral No.3-1	Whey mineral No.3-2	Whey mineral No.3-3	Whey mineral No.3-4	Whey mineral No.4
20 mg%	2.50	5.00	6.50	5.00	7.00	6.25	6.50	6.25
25 mg%	2.00	4.25 (2.00)	5.00	4.75 (2.75)	6.00	5.50	5.75	6.00
30 mg%	-	3.25 (1.75)	4.75 (3.50)	4.50 (2.00)	5.00 (3.75)	5.00	5.25	5.00
35 mg%	-	3.00	4.25 (3.00)	4.00	4.50 (3.25)	4.75 (3.25)	4.50 (3.00)	4.25
40 mg%	-	2.75	4.00 (2.00)	4.00	4.25 (2.00)	4.00 (2.00)	4.25 (2.00)	4.00
45 mg%	-	2.00	2.50	2.50	3.00	2.25	3.50	3.00
50 mg%	-	2.00	2.00	2.00	2.50	2.00	2.00	2.00

Note: The figures in parenthesis represent CTs at an oil-bath temperature of 140 °C.

[0041] When pouring the solution into a sterilizing machine, the CTs with no occurrence of the scorching of protein onto the plate, the precipitation of the sterilization solutions, or the formation of aggregates were 4.00 minutes and 2.00 minutes at 135 and 140 °C, respectively. The Whey mineral Nos. 2, 3-1, 3-2, 3-3, 3-4 and 4 have thus heat resistance.

Example 2

Testing of calcium precipitation

[0042] Centrifuging samples with good heat resistance as shown in Example 1, namely samples of the Whey mineral Nos. 2, 3-1, 3-2, 3-3, 3-4 and 4, at 1,600 g for 10 minutes, the calcium levels of the supernatants were assayed. As a control, a sample with addition of calcium chloride (a soluble calcium salt) was assayed in the same fashion as described above. The results are shown in Table 6.

Table 6

Ca level from whey mineral	Control $CaCl_2$	Whey mineral No. 2	Whey mineral No. 3-1	Whey mineral No. 3-2	Whey mineral No. 3-3	Whey mineral No. 3-4	Whey mineral No. 4
20 mg%	151	148	148	147	148	135	149
30 mg%	160	159	161	160	156	138	158
40 mg%	172	167	165	166	163	137	169
50 mg%	179	175	174	176	170	139	178
							(unit: mg%)

As apparently shown in Table 6, the samples of the Whey mineral Nos. 2, 3-1, 3-2, 3-3 and 4 each at a calcium content of 2 to 8 % by weight retained almost the same level of calcium in the supernatants thereof even after centrifuging, as in the control with the soluble calcium salt. Thus, it is indicated that no precipitation of calcium salts may occur. As shown in the Example 1, the Whey mineral Nos. 2, 3-1, 3-2, 3-3 and 4 also have heat resistance, so that it can be said that these minerals are preferable calcium-supplementing sources.

Example 3

[0043] Dissolving the Whey mineral No.3-2 (455 g) and a defatted powdery milk (10.3 kg) in water (89 kg) at 23 °C, the resulting mixture was homogenized at two steps (a first step at 100 kg/cm^2 and a second step at 50 kg/cm^2). The homogenized solution was sterilized under the conditions of 140 °C for 4 seconds by means of a UHT manufactured by Iwai Machinery Industry, Co. Ltd. Then, no pressure decrease or the like possibly due to the scorching of the protein on the sterilizing machine was observed. Three months after the storage of the resulting sterilized solution at ambient temperature in an aseptic state, no occurrence of rough feeling or abnormal taste or odor was observed.

[0044] Centrifuging at 1,600 g for 10 minutes the calcium-supplemented milk drink immediately after the sterilization, the calcium level in the supernatant was assayed together with the precipitate weight in percent. The same assay was conducted three months after the drink was left to stand at ambient temperature. For a control, calcium chloride was used in place of the whey mineral (with no sterilization treatment). The results are shown in Table 7.

Example 4

[0045] Dissolving the Whey mineral No.2 (71 g) and a defatted powdery milk (1.03 kg) in water (8.9 kg) at 24 °C, the resulting mixture was treated in the same manner as in Example 3. No pressure decrease or the like possibly due to the scorching of the protein on the sterilizing machine was observed. Three months after the resulting solution was left to stand at ambient temperature, no occurrence of rough feeling or abnormal taste or odor was observed.

[0046] Centrifuging the resulting product in the same manner as in Example 3, immediately after the sterilization and three months after the product was left to stand at ambient temperature, the calcium level in the supernatant was assayed together with the precipitate weight in percent. The results are shown concurrently in Table 7.

Table 7

	Example 3	Example 4	Control $CaCl_2$
Ca level immediately after sterilization	155 mg %	157 mg %	159 mg %
Precipitate weight immediately after sterilization	0.15 wt %	0.20 wt %	0.85 wt %
Ca level after 3-month simple storage at ambient temperature	152 mg %	154 mg %	----
Precipitate weight after 3-month simple storage at ambient temperature	0.21 wt %	0.23 wt %	----

[0047] As apparently shown in Table 7, the samples using the whey minerals of the Production Examples have less precipitate of calcium salts, and the levels of the precipitate do not change after the long-term storage.

[0048] Five skilled panelists tested the flavor of the calcium-supplemented milk drinks of the Examples 3 and 4 of the present invention and the flavor of the control using calcium chloride. Consequently, all the panelists made judgment such that the products of the present invention had stronger body and better flavor than those of the control.

Example 5

[0049] As in Examples 3 and 4, the same experiments were conducted using commercially available whey minerals. When the Ca concentration from the whey minerals and the Ca concentration from defatted powdery milk were given at 30 mg% and 130 mg%, respectively, the coagulation time at 135 °C and the Ca concentration in the supernatant after centrifuge were determined, as shown in Table 8.

[0050] "Versapro E" of a Ca content of 5% had excellent heat resistance with less Ca precipitate, compared with other whey minerals. No abnormal sterilizing machine, no occurrence of rough feeling phenomenon under storage, or no abnormal taste or odor was observed; or almost no Ca precipitation was observed under storage.

Table 8

Composition and coagulation time of commercially available whey minerals and Ca concentration in the supernatant after centrifuge			
	Ca content (%)	Coagulation time (min.)	Ca concentration in supernatant (mg%)
Meiji whey powder (Meiji Milk Products Co., Ltd.)	1.2	2.00	156
Suval (Valio Ltd.)	1.6	2.75	155
Versapro E (DAVISCO Foods International, Inc.)	5.0	5.50	158
Capolac (MD Foods Ingredients amba)	18.0	6.00	138
Mini flow 34 Plus (FDA Co., Ltd)	30.0	5.75	135

Effects of the Invention

[0051] In accordance with the present invention, it is provided a calcium-supplemented milk drink, having good flavor with no precipitation of calcium salts or being provided with attractive flavor, and additionally being capable of passing through the sterilization process to be prepared into a commercially aseptic state.

Claims

1. A process for producing a calcium-supplemented cow's milk drink, said process, which comprises using whey as a Ca source, obtaining a mixture of whey and cow's milk then sterilizing said mixture, being characterized in that it comprises the following steps :

   (a) whey is subjected to protein removal and lactose removal,
   (b) the resulting whey is subjected to pH adjustment to 6.0-9.0,
   (c) the resulting whey is further subjected to ultrafiltration or microfiltration to obtain a concentrate,

(d) the concentrate thus obtained is subjected to drying to obtain a whey mineral having a calcium content of 2-8 % by weight,
(e) the whey mineral thus obtained is added to cow's milk to form a calcium-supplemented cow's milk drink so that the amount of calcium supplied by the whey mineral represents 10-40 mg % therein (10-40 mg Ca for 100 g calcium-supplemented cow's milk drink), and
(f) the calcium-supplemented cow's milk drink thus obtained is subjected to pH adjustment to 6.0-7.5,

whereby the calcium-supplemented cow's milk drink thus obtained is heat resistant with no precipitation of Ca salts and stands :
(g) sterilization as a final step.

2. A process for producing a calcium-supplemented cow's milk drink, said process, which comprises using whey as a Ca source, obtaining a mixture of whey and cow's milk then sterilizing said mixture, being characterized in that it comprises the following steps :

(a) whey is provided,
(b) the whey thus provided is subjected to protein removal,
(c) the resulting whey is subjected to pH adjustment to 6.0-9.0,
(d) the resulting whey is further subjected to centrifuging to obtain a precipitate,
(e) the precipitate thus obtained is subjected to drying to obtain a whey mineral having a calcium content of 2-8 % by weight,
(f) the whey mineral thus obtained is added to cow's milk to form a calcium-supplemented cow's milk drink so that the amount of calcium supplied by the whey mineral represents 10-40 mg % therein (10-40 mg Ca for 100 g calcium-supplemented cow's milk drink), and
(g) the calcium-supplemented cow's milk drink thus obtained is subjected to pH adjustment to 6.0-7.5,

whereby the calcium-supplemented cow's milk drink thus obtained is heat resistant with no precipitation of Ca salts and stands :
(h) sterilization as a final step.

3. A process for producing a calcium-supplemented cow's milk drink, said process, which comprises using whey as a Ca source, obtaining a mixture of whey and cow's milk then sterilizing said mixture, being characterized in that it comprises the following steps :

(a) whey is provided,
(b) the whey thus provided is subjected to pH adjustment to 6.0-9.0,
(c) the resulting whey is further subjected to centrifuging to obtain a precipitate,
(d) the precipitate thus obtained is subjected to drying to obtain a whey mineral having a calcium content of 2-8 % by weight,
(e) the whey mineral thus obtained is added to cow's milk to form a calcium-supplemented cow's milk drink so that the amount of calcium supplied by the whey mineral represents 10-40 mg % therein (10-40 mg Ca for 100 g calcium-supplemented cow's milk drink), and
(f) the calcium-supplemented cow's milk drink thus obtained is subjected to pH adjustment to 6.0-7.5,

whereby the calcium-supplemented cow's milk drink thus obtained is heat resistant with no precipitation of Ca salts and stands :
(g) sterilization as a final step.

4. The process according to claim 1, wherein the protein removal in step (a) is carried out by ultrafiltration or cation exchange.

5. The process according to claim 2, wherein the protein removal in step (b) is carried out by ultrafiltration or cation exchange.

6. The process according to claim 1, wherein the cow's milk is selected from the group consisting of partially defatted milk, defatted milk, defatted powdered milk, reconstituted milk and processed milk.

7. The process according to claim 2, wherein the cow's milk is selected from the group consisting of partially defatted

milk, defatted milk, defatted powdered milk, reconstituted milk and processed milk.

8. The process according to claim 3, wherein the cow's milk is selected from the group consisting of partially defatted milk, defatted milk, defatted powdered milk, reconstituted milk and processed milk.

Patentansprüche

1. Verfahren zur Herstellung von mit Kalzium angereicherten Milchgetränken, unter der Verwendung von Molke als Ca-Quelle, mit
der Erzeugung einer Mischung aus Molke und Milch mit anschließender Sterilisation der Milch,
dadurch gekennzeichnet, daß
es die folgenden Schritte umfaßt:

    a) Protein- und Lactosetrennung von Molke,
    b) anschließende Einstellung des PH-Werts der Molke auf 6.0 - 9.0,
    c) anschließende Konzentratbildung durch Ultrafiltrierung oder Mikrofiltrierung der Molke,
    d) anschließendes Trocknen des Konzentrats zur Mineralbildung mit einem Kalziumgehalt von 2 - 8 Gewichtsprozent,
    e) Zusetzen des resultierenden Molkeminerals zu Milch zur Erzeugung eines Kalzium-angereicherten Milchgetränks mit einem Kalziumanteil des Molkeminerals von 10 - 40 mg % (10 - 40 mg Ca pro 100 g Kalzium-angereichertem Milchgetränk), und
    f) anschließende Einstellung des pH-Werts des Kalzium-angereicherten Milchgetränks auf 6.0 - 7.5,
    wobei das Kalzium-angereicherte Milchgetränk wärmebeständig ist ohne Ca-Salze auszuscheiden und sterilisationsfähig ist:
    g) abschließende Sterilisation

2. Verfahren zur Herstellung von mit Kalzium angereicherten Milchgetränken unter der Verwendung von Molke als Ca-Quelle, mit der Erzeugung einer Mischung aus Molke und Milch mit anschließender Sterilisation der Milch, dadurch gekennzeichnet, daß es die folgenden Schritte umfaßt:

    a) Molkezubereitung,
    b) anschließende Proteintrennung von der Molke,
    c) anschließende Einstellung des pH-Werts der Molke auf 6.0 - 9.0,
    d) anschließendes Schleudern der Molke mit Bildung einer Ausscheidung,
    e) anschließende Trocknung der Ausscheidung zur Mineralbildung mit einem Kalziumgehalt von 2 - 8 Gewichtsprozent,
    f) Zusetzen des resultierenden Minerals zu Milch zur Erzeugung eines Kalzium-angereicherten Milchgetränks mit einem Kalziumanteil des Molkeminerals von 10 - 40 mg % (10 - 40 mg Ca pro 100 g Kalzium-angereicherten Milchgetränkes) und
    g) anschließende Einstellung des pH-Werts des Kalzium-angereicherten Milchgetränks auf 6.0 - 7.5,
    wobei das Kalzium-angereicherte Milchgetränk wärmebeständig ist ohne Ca-Salze auszuscheiden und sterilisationsfähig ist:
    h) abschließende Sterilisation

3. Verfahren zur Herstellung von mit Kalzium angereicherten Milchgetränken unter der Verwendung von Molke als Ca Quelle, der Erzeugung einer Mischung aus Molke und Milch mit anschließender Sterilisation der Milch, dadurch gekennzeichnet, daß es die folgenden Schritte umfaßt:

    a) Molkezubereitung,
    b) anschließende Einstellung des pH-Werts der Molke auf 6.0 - 9.0,
    c) anschließendes Schleudern der Molke mit Bildung einer Ausscheidung
    d) anschließende Trocknung der Ausscheidung zur Mineralbildung mit einem Kalziumgehalt von 2 - 8 Gewichtsprozent,
    e) Zusetzen des resultierenden Minerals zu Milch zur Erzeugung eines Kalzium-angereicherten Milchgetränks mit einem Kalziumanteil des Molkeminerals von 10 - 40 mg % (10 - 40 mg Ca pro 100 g Kalzium-angereicherten Milchgetränkes), und
    f) anschließende Einstellung des pH-Werts des Kalzium-angereicherten Milchgetränks auf 6.0 - 7.5,

wobei das Kalzium-angereicherte Milchgetränk wärmebeständig ist ohne Ca-Salze auszuscheiden und sterilisationsfähig ist:
g) abschließende Sterilisation

4. Verfahren nach Anspruch 1, wobei die Proteintrennung in Schritt (a) mittels Ultrafiltrierung oder Kationaustausch durchgeführt wird.

5. Verfahren nach Anspruch 2, wobei die Proteintrennung in Schritt (b) mittels Ultrafiltrierung oder Kationaustausch durchgeführt wird.

6. Verfahren nach Anspruch 1, wobei die Milch ausgewählt ist aus der Gruppe bestehend aus teilweise entfetteter Milch, entfetteter Milch, Milchpulver aus entfetteter Milch, zurückgeführter Milch und behandelter Milch.

7. Verfahren nach Anspruch 2, wobei die Milch ausgewählt ist aus der Gruppe bestehend aus teilweise entfetteter Milch, entfetteter Milch, Milchpulver aus entfetteter Milch, zurückgeführter Milch und behandelter Milch.

8. Verfahren nach Anspruch 3, wobei die Milch ausgewählt ist aus der Gruppe bestehend aus teilweise entfetteter Milch, entfetteter Milch, Milchpulver aus entfetteter Milch, zurückgeführter Milch und behandelter Milch.

Revendications

1. Procédé de production d'une boisson à base de lait de vache complémenté en calcium, ledit procédé qui comprend l'utilisation de petit lait en tant que source de Ca, l'obtention d'un mélange de petit-lait et de lait de vache, puis la stérilisation dudit mélange, étant caractérisé en ce qu'il comprend les étapes suivantes :

   (a) le petit-lait est soumis à une élimination des protéines et à une élimination du lactose,
   (b) le petit-lait résultant est soumis à un ajustement du pH à 6,0-9,0,
   (c) le petit-lait résultant est, en outre, soumis à une ultrafiltration ou à une microfiltration pour obtenir un concentré,
   (d) le concentré ainsi obtenu est soumis à un séchage pour obtenir un petit-lait minéral ayant une teneur en calcium de 2-8% en poids,
   (e) le petit-lait minéral ainsi obtenu est ajouté à du lait de vache pour former une boisson à base de lait de vache complémenté en calcium, de sorte que la proportion de calcium fournie par le petit-lait minéral y représente 10-40% (10-40 mg de Ca pour 100 g de boisson à base de lait de vache complémenté en calcium), et
   (f) la boisson à base de lait de vache complémenté en calcium ainsi obtenue est soumise à un ajustement du pH à 6,0-7,5,

   afin que la boisson à base de lait de vache complémenté en calcium ainsi obtenue soit thermo-résistante sans aucune précipitation de sels de Ca ni aucun dépôt ;
   (g) une stérilisation en tant qu'étape finale.

2. Procédé de production d'une boisson à base de lait de vache complémenté en calcium, ledit procédé qui comprend l'utilisation de petit-lait comme source de Ca, l'obtention d'un mélange de petit-lait et de lait de vache, puis la stérilisation dudit mélange, étant caractérisé en ce qu'il comprend les étapes suivantes :

   (a) se munir de petit-lait,
   (b) le petit-lait ainsi obtenu est soumis à une élimination des protéines,
   (c) le petit-lait résultant est soumis à un ajustement du pH à 6,0-9,0,
   (d) le petit-lait résultant est, en outre, soumis à une centrifugation pour obtenir un précipité,
   (e) le précipité ainsi obtenu est soumis à un séchage pour obtenir du petit-lait minéral ayant une teneur en calcium de 2-8% en poids,
   (f) le petit-lait minéral ainsi obtenu est ajouté à du lait de vache pour former une boisson à base de lait de vache complémenté en calcium, de sorte que la proportion de calcium fournie par le petit-lait minéral y représente 10-40% (10-40 mg de Ca pour 100 g de boisson à base de lait de vache complémenté en calcium), et
   (g) la boisson à base de lait de vache complémenté en calcium ainsi obtenue est soumise à un ajustement du pH à 6,0-7,5,

afin que la boisson à base de lait de vache complémenté en calcium ainsi obtenue soit thermo-résistante sans aucune précipitation de sels de Ca ni aucun dépôt ;

(h) une stérilisation en tant qu'étape finale.

3. Procédé de production d'une boisson à base de lait de vache complémenté en calcium, ledit procédé qui comprend l'utilisation de petit lait comme source de Ca, l'obtention d'un mélange de petit-lait et de lait de vache, puis la stérilisation dudit mélange, étant caractérisé en ce qu'il comprend les étapes suivantes :

(a) se munir de petit-lait,
(b) le petit-lait ainsi obtenu est soumis à un ajustement du pH à 6,0-9,0,
(c) le petit-lait résultant est, en outre, soumis à une centrifugation pour obtenir un précipité,
(d) le précipité ainsi obtenu est soumis à un séchage pour obtenir du petit-lait minéral ayant une teneur en calcium de 2-8% en poids,
(e) le petit-lait minéral ainsi obtenu est ajouté à du lait de vache pour former une boisson à base de lait de vache complémenté en calcium, de sorte que la proportion de calcium fournie par le petit-lait minéral y représente 10-40% (10-40 mg de Ca pour 100 g de boisson à base de lait de vache complémenté en calcium), et
(f) la boisson à base de lait de vache complémenté en calcium ainsi obtenue est soumise à un ajustement du pH à 6,0-7,5,

afin que la boisson à base de lait de vache complémenté en calcium ainsi obtenue soit thermo-résistante sans aucune précipitation de sels de Ca ni aucun dépôt ;

(g) une stérilisation en tant qu'étape finale.

4. Procédé selon la revendication 1, dans lequel l'élimination des protéines dans l'étape (a) est réalisée par ultrafiltration ou par échange de cations.

5. Procédé selon la revendication 2, dans lequel l'élimination des protéines dans l'étape (b) est réalisée par ultrafiltration ou par échange de cations.

6. Procédé selon la revendication 1, dans lequel le lait de vache est choisi dans le groupe constitué par le lait demi-écrémé, le lait écrémé, le lait écrémé en poudre, le lait reconstitué et le lait traité.

7. Procédé selon la revendication 2, dans lequel le lait de vache est choisi dans le groupe constitué par le lait demi-écrémé, le lait écrémé, le lait écrémé en poudre, le lait reconstitué et le lait traité.

8. Procédé selon la revendication 3, dans lequel le lait de vache est choisi dans le groupe constitué par le lait demi-écrémé, le lait écrémé, le lait écrémé en poudre, le lait reconstitué et le lait traité.